Progress with capping layer and optics refurbishment at RIT

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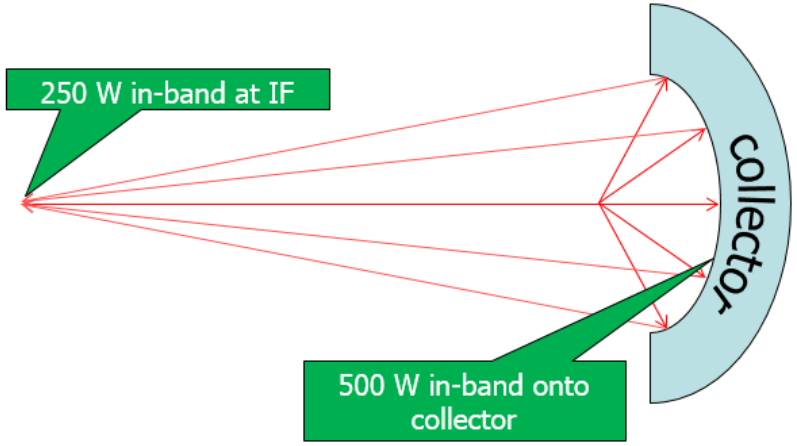
Outline

- Introduction
- Capping layer
  - Deposition
  - EUV exposure
  - Ellipsometry, XPS, EUV reflectivity
- Refurbishment
  - Sn removal
  - Mo/Si removal: wet etching, plasma etching
- Conclusion

Rigaku

ENVIRONMENT NEAR AN LPP SOURCE IS HARSH:

- High thermal load (1 – 10 W/cm^2)
- EUV-assisted oxidation
- EUV-assisted carbonization
- Fast ions & neutrals
  (sputtering & implantation)
- Sn vapor & liquid deposition



For a collector that reflects ~50% of in-band radiation to produce 250W at the IF, requires average intensity of about 300 mW/cm^2 onto the multilayer surface

MOTIVATION FOR THIS STUDY:

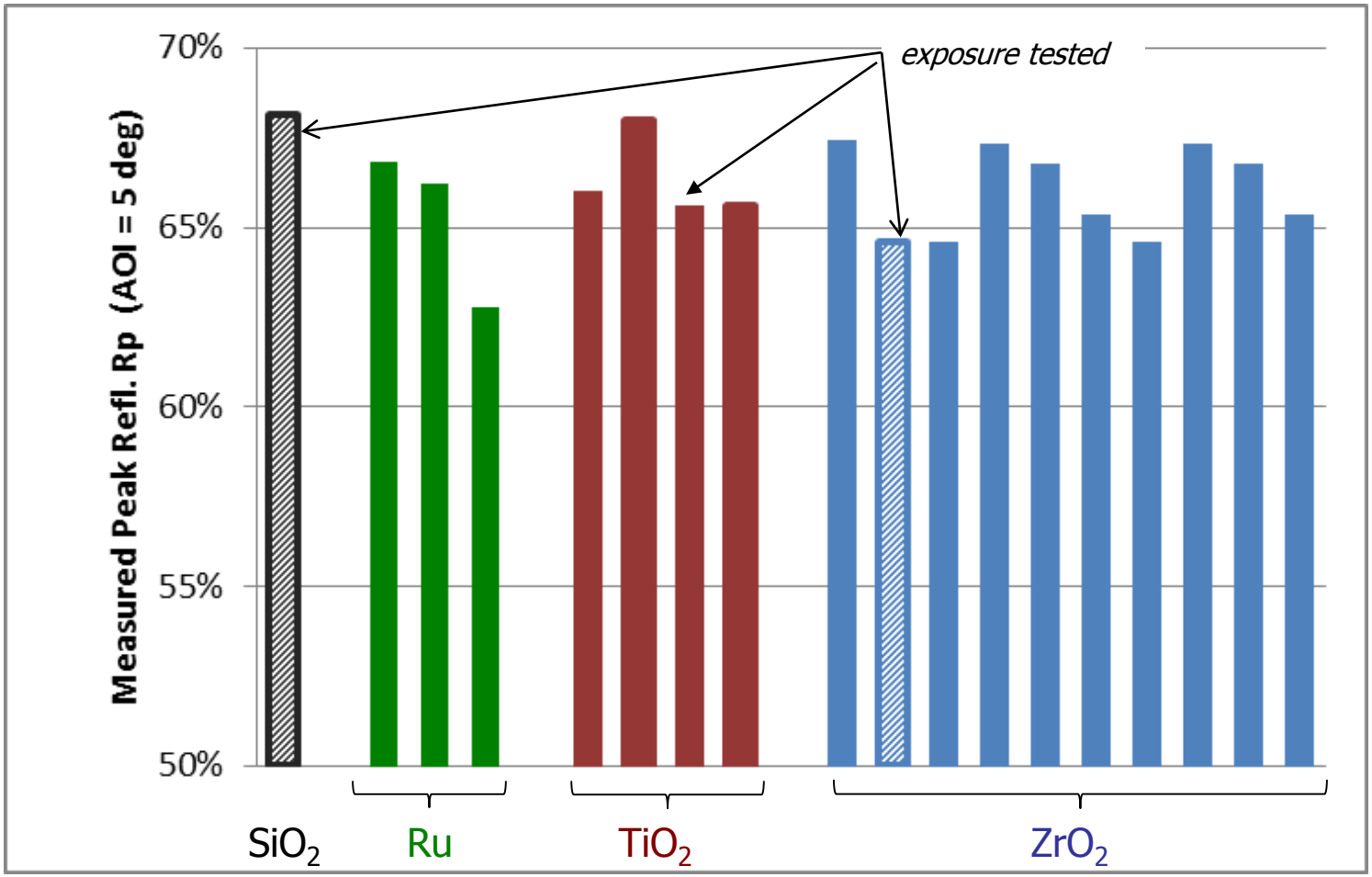
i. Resistance to oxidation occurring from background water vapor

ii. Resistance to damage from Sn-removal/cleaning process(es)

iii. Collector' ML coating repairing

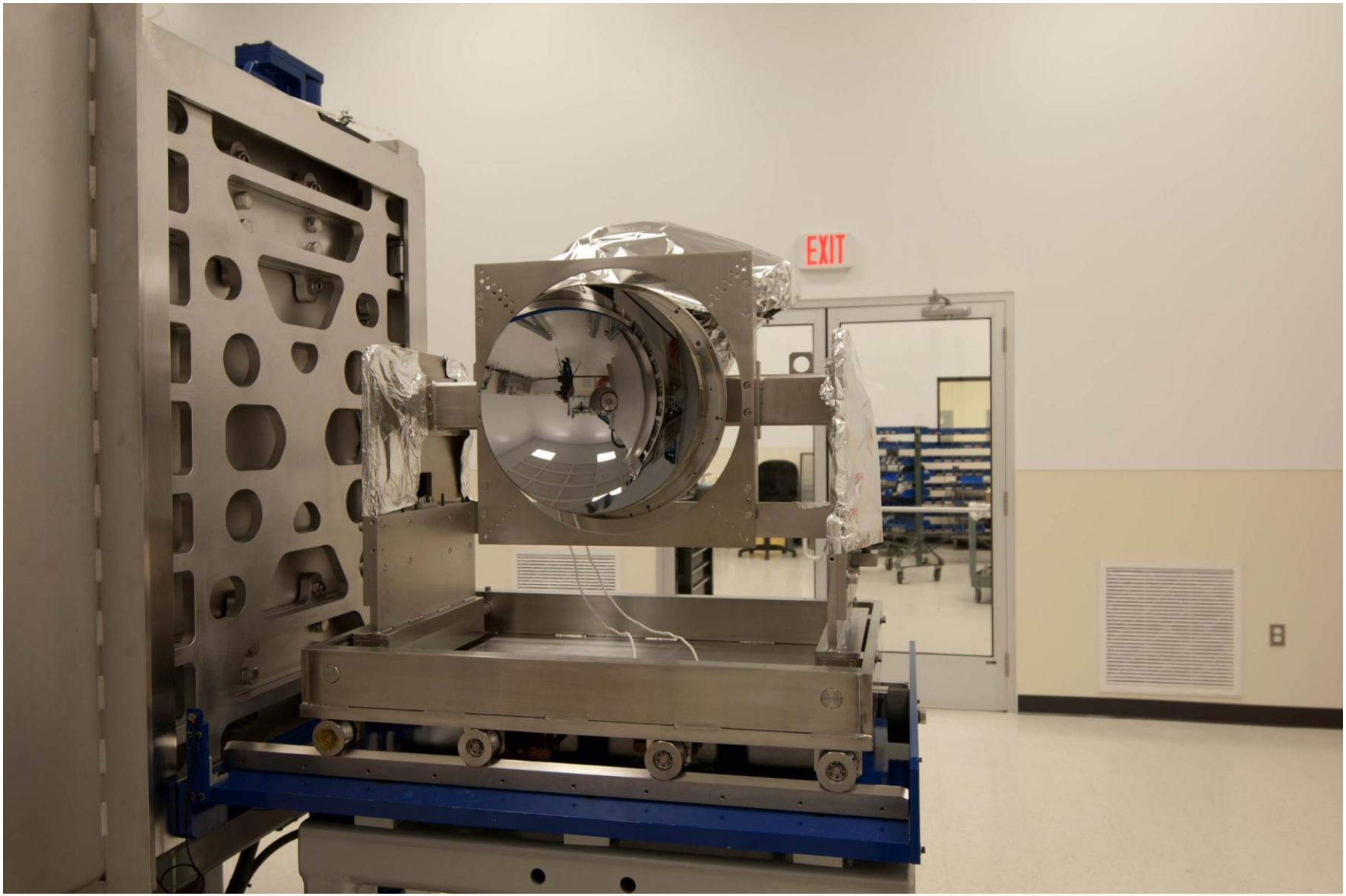
Deposited capping layers

An array of pre-cap and capping layer oxide thicknesses were deposited onto Mo/Si and Mo/B_4C/Si multilayers

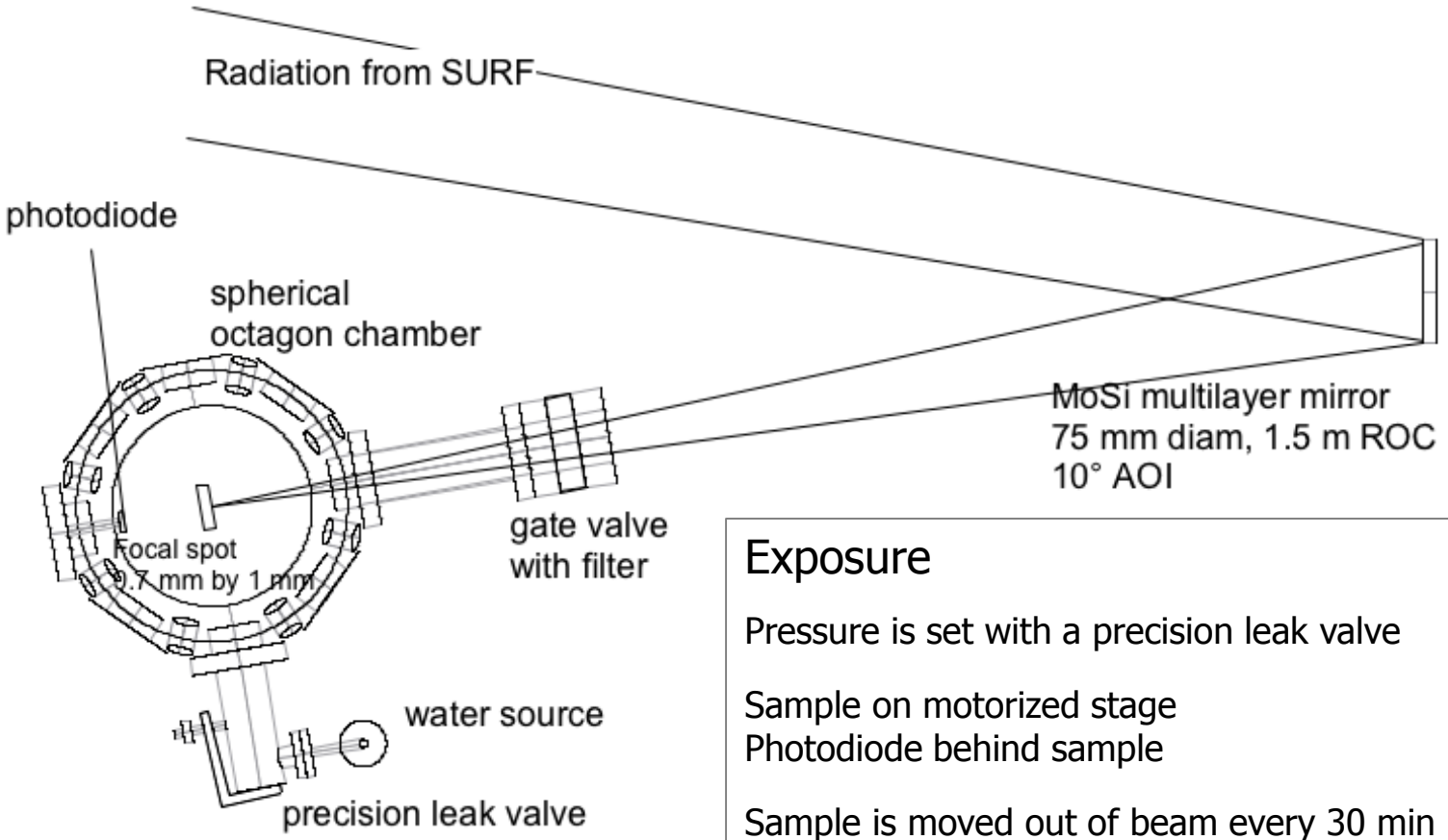


ING2 deposition system

Rotary cart for a large optics



NIST EUV Exposure Apparatus



Exposure

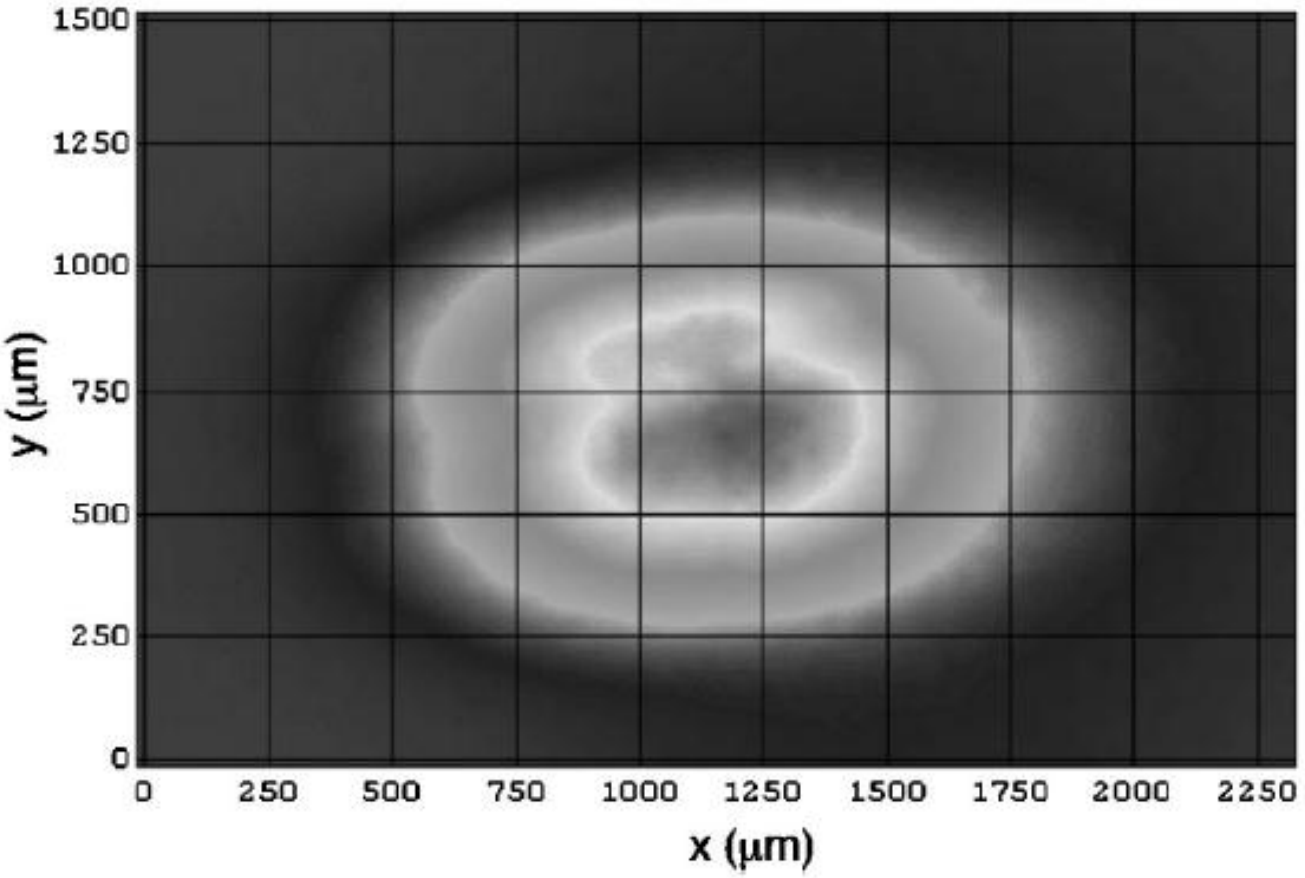
Pressure is set with a precision leak valve

Sample on motorized stage
Photodiode behind sample

Sample is moved out of beam every 30 min
Photocurrent and SURF current measured
SURF current monitored every few seconds
Maximum drift in ratio 2%

Sample is exposed to a specified fluence

Illumination spot



Average power of 1.75 mW with an average intensity of ~ 138 mW/cm^2

Samples analysis methods (NIST)

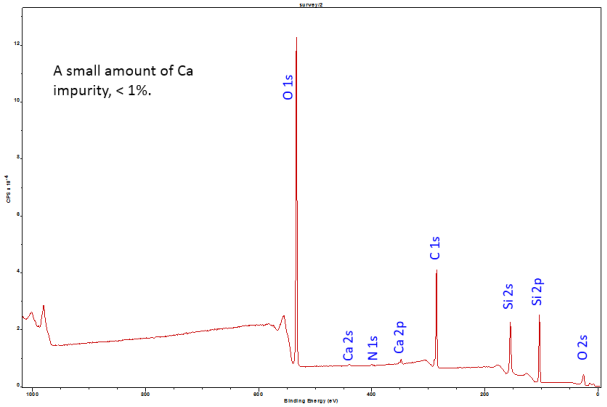
XPS – Oxidation: oxides, sub-oxides, stoichiometry, impurities

Ellipsometry – location and size of exposed area, relative scale of changes after exposure

Reflectometry – Reflectivity loss

XPS: stoichiometry - SiO_2 & TiO_2

SiO_2



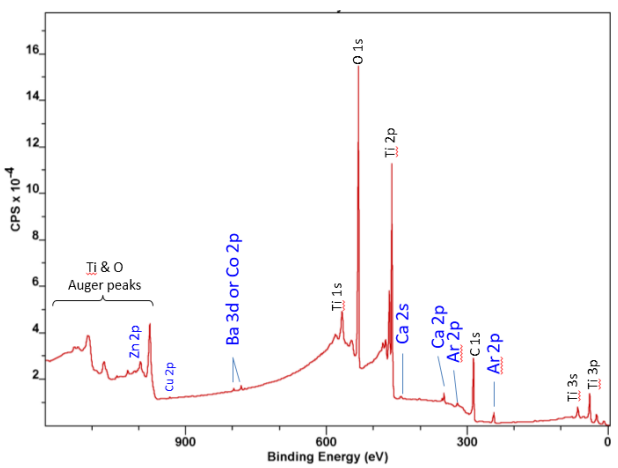
Spot 1 **O:Si = 2.0**

Block Id	Element (Molecule)	Atomic Percentage
O 1s/2	O	44
Si 2s/4	Si	22
C 1s/3	C	33

Spot 2 **O:Si = 1.8**

Block Id	Element (Molecule)	Atomic Percentage
O 1s/7	O	46
Si 2s/9	Si	25
C 1s/8	C	29

TiO_2

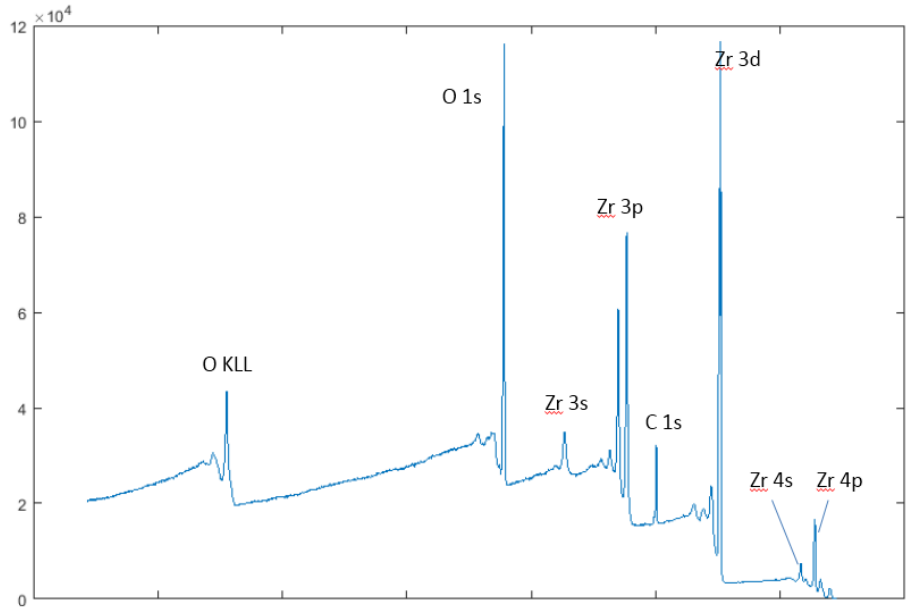


Ratio	TiO2
O1s(oxide) / Ti	1.9
O1s(oxide + carbonate) / Ti	2.1

Carbonate formation does not seem to play a role in TiO2 deposition. The Ti spectra suggest the ~5% of "missing" oxide is likely due to sub-oxide.

Both Si and Ti show practically a full oxidation

XPS: stoichiometry – ZrO_2



- **All Zr is bound to O**
- **~15% - 25% of zirconium carbonate present**

Spot 1

Block Id	Name	Atomic Percentage
Zr 3d/5	Zr 3d	24
O 1s/2	O 1s (ZrO2)	41
O 1s/2	O 1s (ZrCO3)	10
C 1s/4	C 1s	22
C 1s/4	C 1s (ZrCO3)	2

Spot 2

Block Id	Name	Atomic Percentage
Zr 3d/11	Zr 3d	25
O 1s/8	O 1s (ZrO2)	38
O 1s/8	O 1s (ZrCO3)	13
C 1s/10	C 1s	22
C 1s/10	C 1s (ZrCO3)	2

Confirm that all Zr is bound to O:

$$\left(\begin{matrix}\text{Fraction of Zr}\\ \text{bound to O}\end{matrix}\right) = \frac{O(ZrO2)/2 + O(ZrCO3)/3}{Zr}$$

Spot 1: $\frac{41/2 + 10/3}{24} = 99\%$

Spot 2: $\frac{38/2 + 13/3}{25} = 93\%$

$$\left(\begin{matrix}\textbf{Fraction of Zr}\\ \textbf{in } \mathbf{ZrO_2}\end{matrix}\right) = \frac{O(ZrO2)/2}{Zr}$$

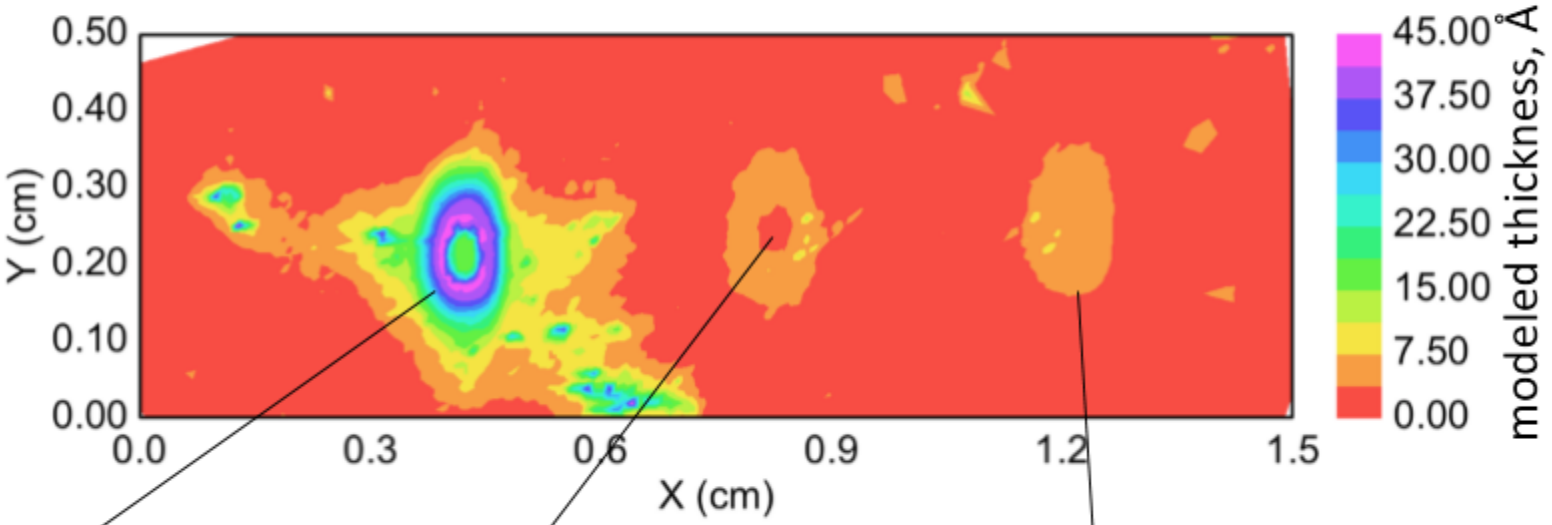
Spot 1: $\frac{41/2}{24} = \mathbf{85\%}$

Spot 2: $\frac{38/2}{25} = \mathbf{76\%}$

Ellipsometry: TiO2 samples

Exposure samples evaluated with spectroscopic ellipsometry at NIST to identify changes in structure from total dosage & background H_2O pressure

TiO_2 capping layer sample



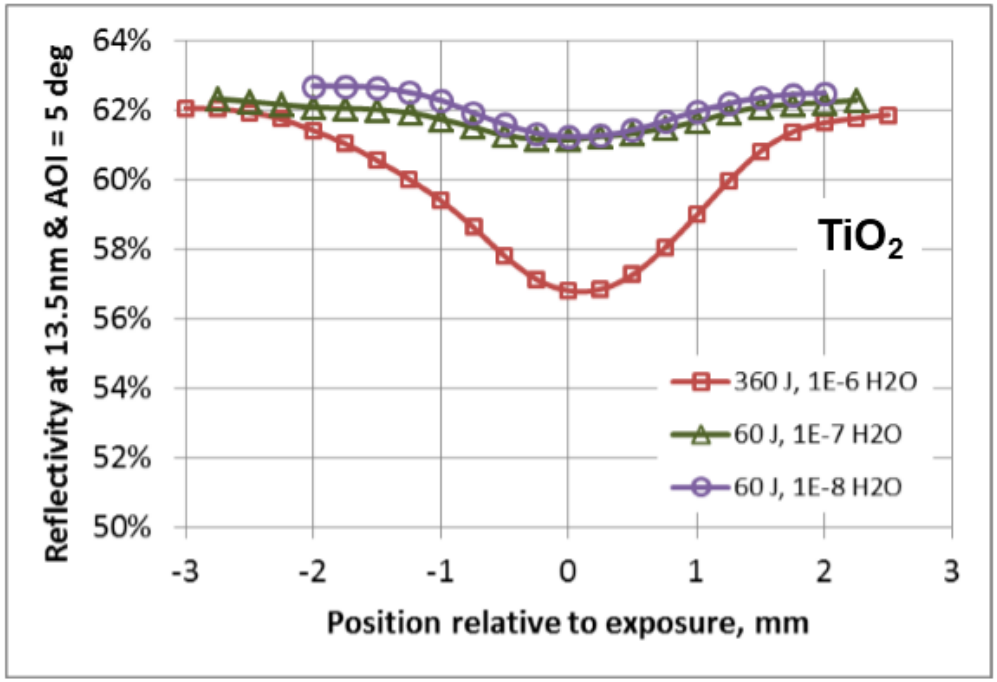
H_2O 1x10^{-6} Torr
55 hours
360 J dose

H_2O 1x10^{-7} Torr
10hours
60J dose

H_2O 1x10^{-8} Torr
10 hours
60J dose

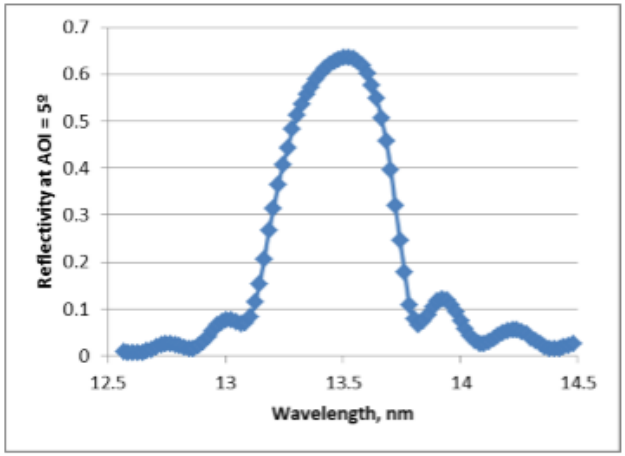
EUV reflectivity: TiO_2

Line-scans at λ = 13.45 nm through the center of each exposure†



Exposure	Refl. Loss
360 J, 1E-6 H2O	5.2% abs. units
60 J, 1E-8 H2O	1.3% abs. units
60 J, 1E-7 H2O	1.2% abs. units

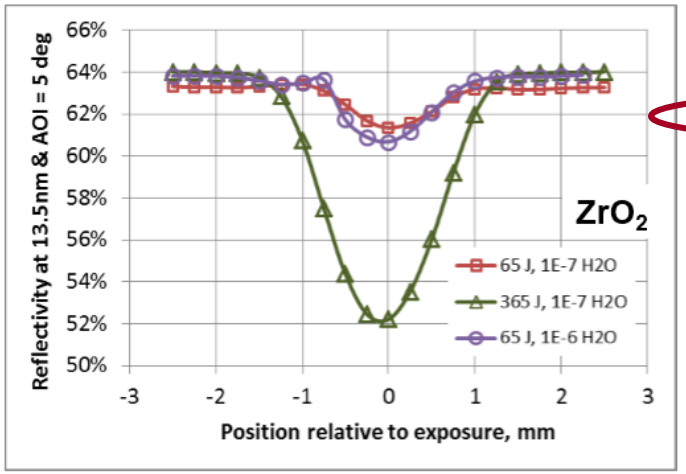
5.2% loss



†Rp is 63.5% at 13.524nm

EUV reflectivity: ZrO_2 improved

Line-scans at λ = 13.4nm through the center of each exposure

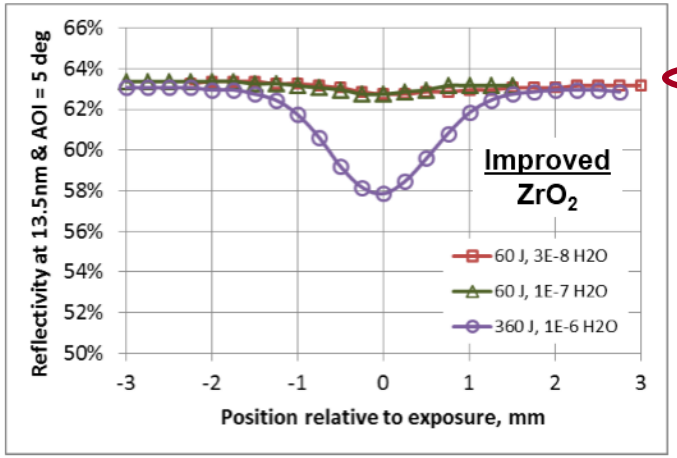


Exposure	Refl. Loss
65 J, 1E-7 H2O	1.9% abs. units
65 J, 1E-6 H2O	3.0% abs. units
365 J, 1E-6 H2O	11.0% abs. units

February' result

11% loss

Line-scans at λ = 13.68nm through the center of each exposure



Exposure	Refl. Loss
60 J, 3E-8 H2O	0.4% abs. units
360 J, 1E-6 H2O	5.2% abs. units
60 J, 1E-7 H2O	0.6% abs. units

June' result

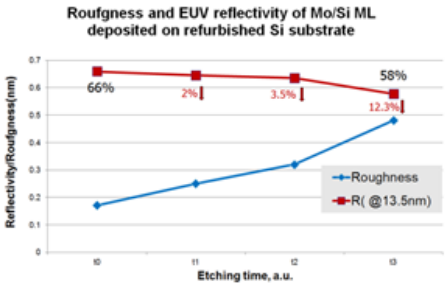
5.2% loss

Improved ZrO_2 capping layer demonstrated ~ 2 times lower EUV reflectivity loss

Old results on refurbishing Mo/Si ML

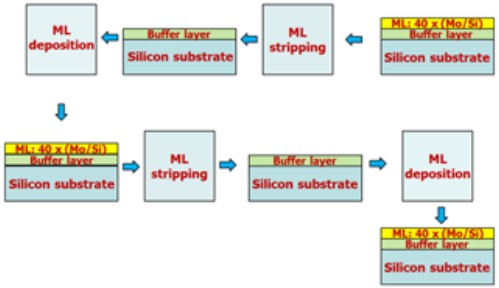
No Buffer layer

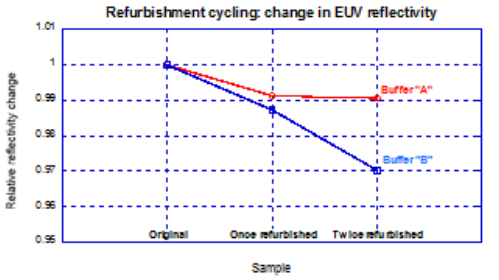
Mo/Si multilayer structure with a cap layer deposited on Si substrate – no buffer (current M1-M5 mirrors delivered to EDEC)



~ 2% loss per cycle with no buffer layer

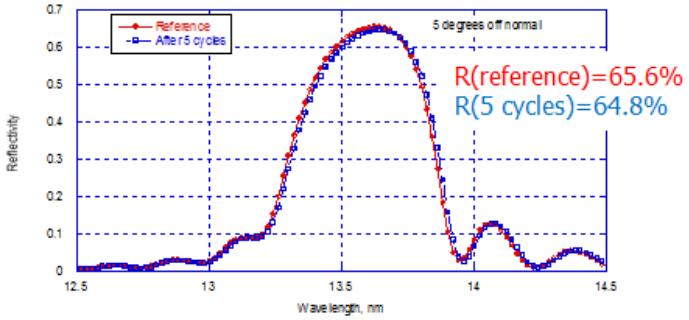
Introducing a buffer layer
(2 cycles - original coating has no buffer layer)

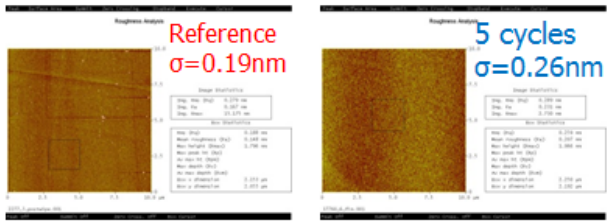




~ 1.9% after 2nd cycle with a buffer layer
(no buffer layer in original coating)

5 cycles with a buffer layer
(original coating has a buffer layer)_





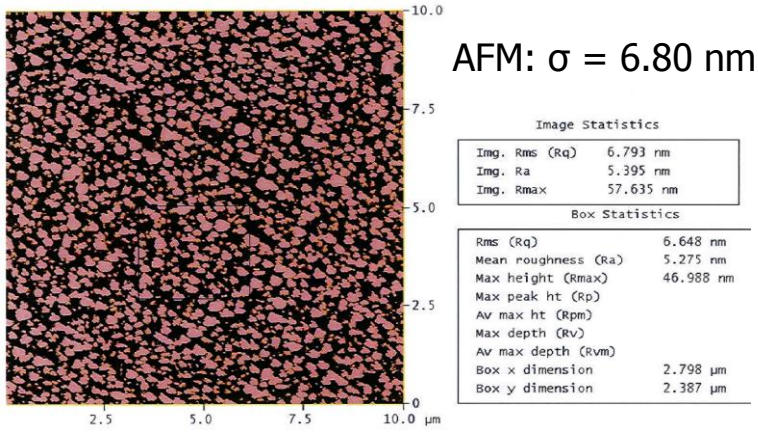
~ 1.2% loss after 5 refurbishment cycles
(original coating has a buffer layer)

Collector refurbishment

- *Large size*
- *Heavy*
- *Curved*
- *Covered by tin*
- *Metal substrate*
- *Cooling fixtures*
- *Presence of a glassy smoothing layer (IRRC)*

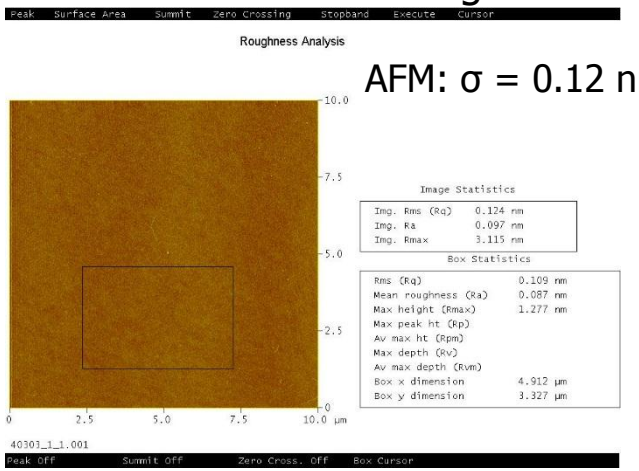
Tin removal (wet etching)

~ 400Å Sn on 4" Si wafer

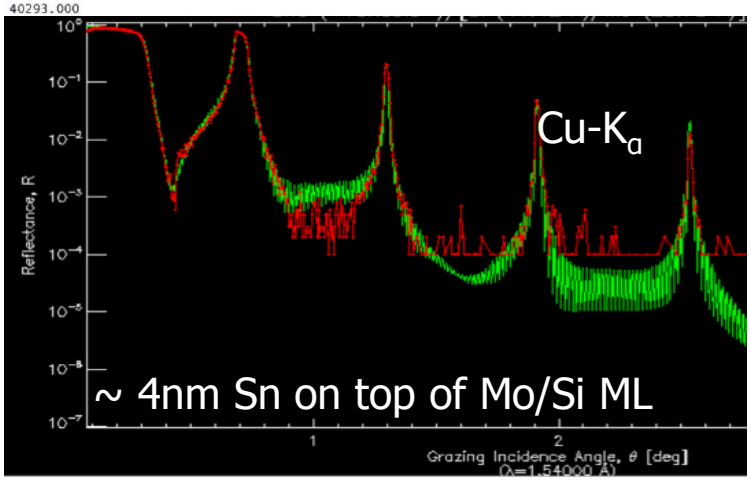


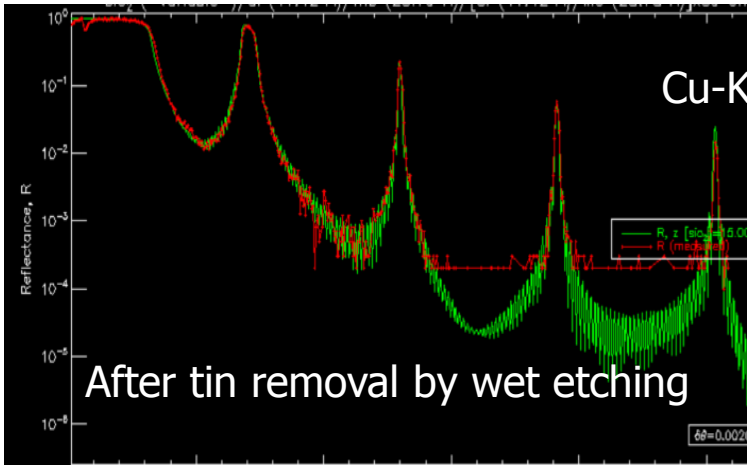
AFM: σ = 6.80 nm rms

After wet etching



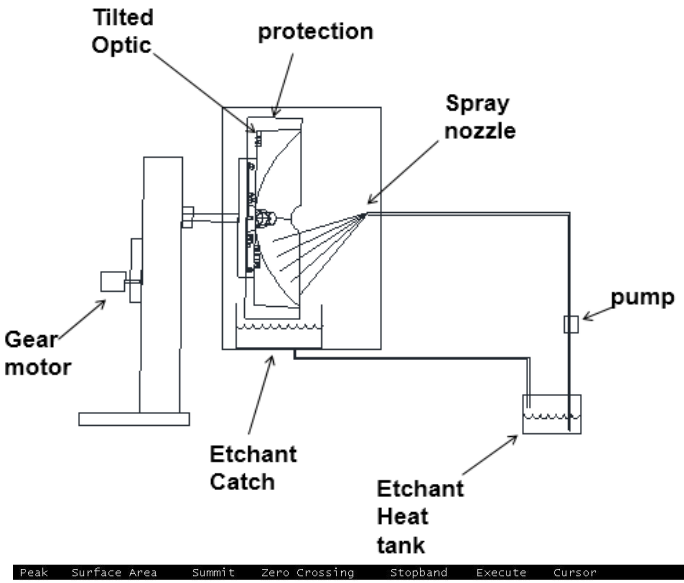
AFM: σ = 0.12 nm rms



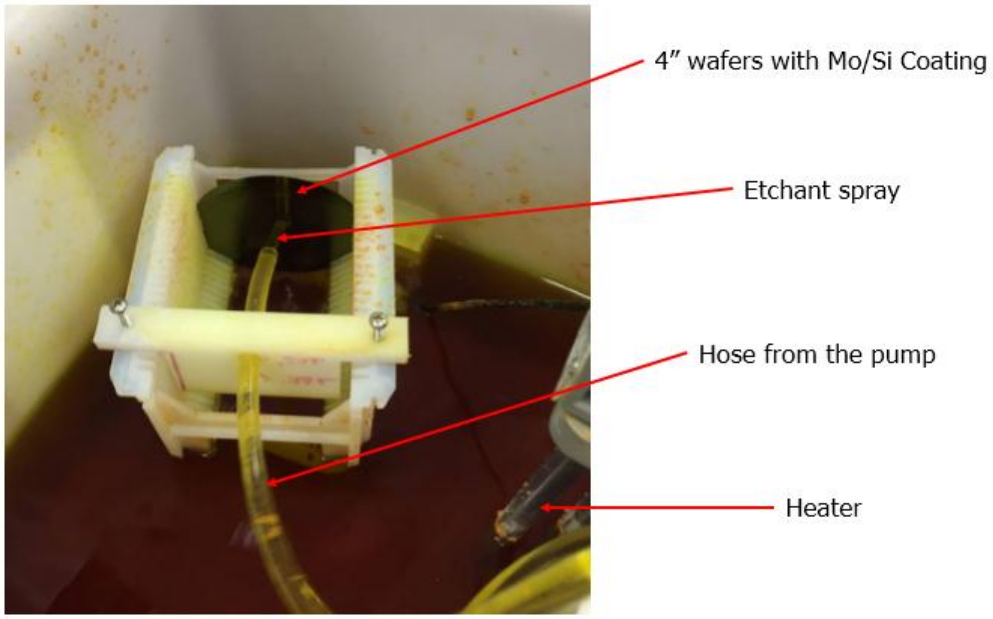


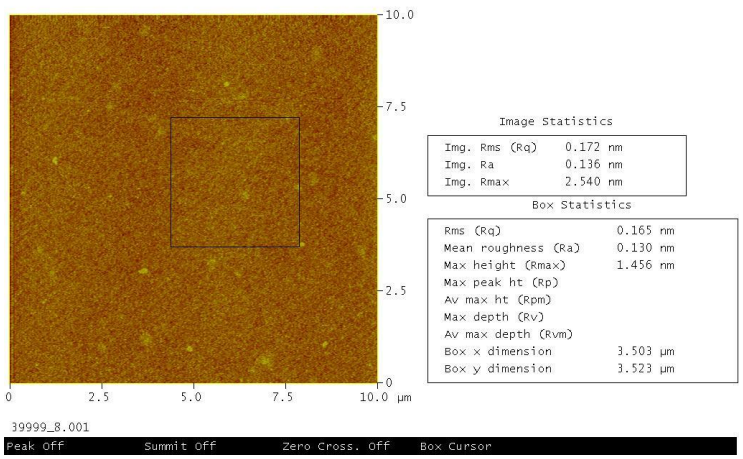
- **Tin layer completely removed by wet etching**
- **No effect on ML performance after tin removal**

Collector wet etching (concept)



Spray etch (pulse pump at 2 pulses per second) in square tank on 4" silicon substrate + 40x ML d=100A Mo/Si





ML was completely removed. Roughness is 0.17 nm rms.

But

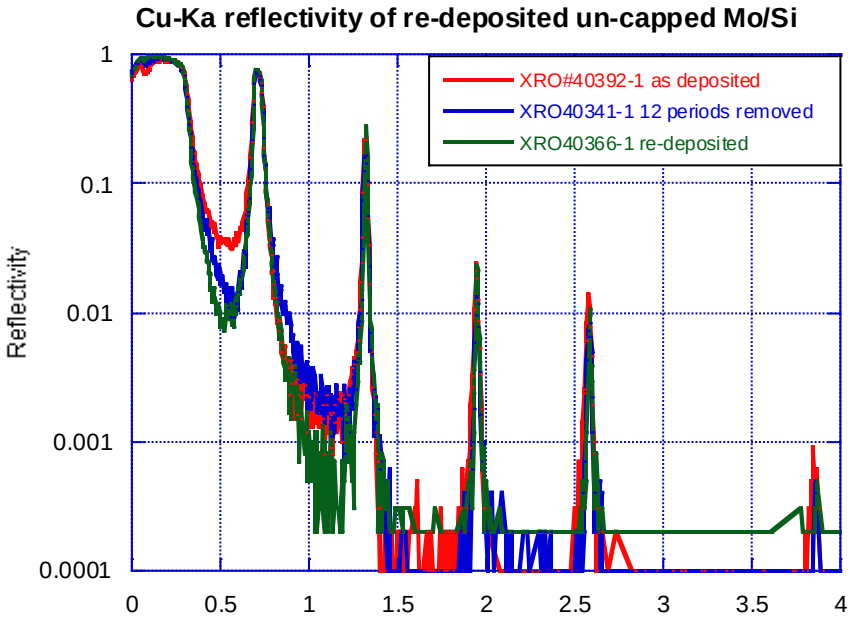
- messy

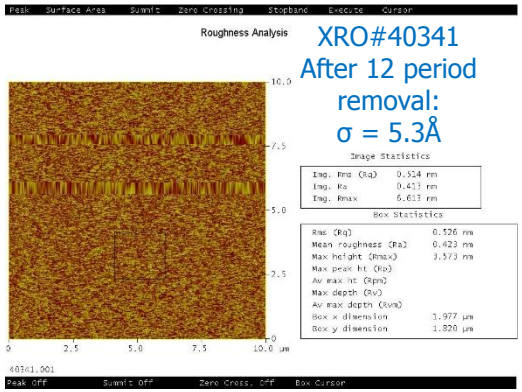
- safety concern

Plasma etching – flat uncapped Mo/Si

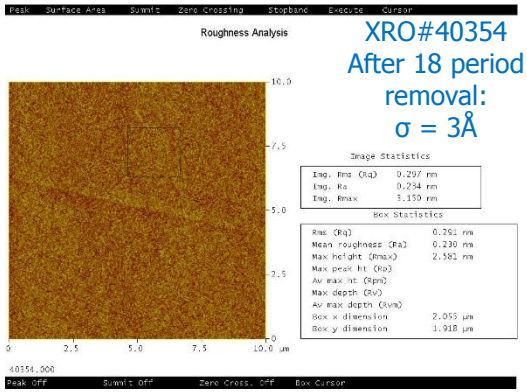
Partial removal and re-deposition

1. 40 periods of Mo/Si deposited
2. 12 periods (XRO#40341) and 18 periods (XRO#40354) removed by a plasma etching
3. 40 periods (XRO#40366 on XRO#40341) and 60 periods (XRO#40359 on XRO#40354) deposited

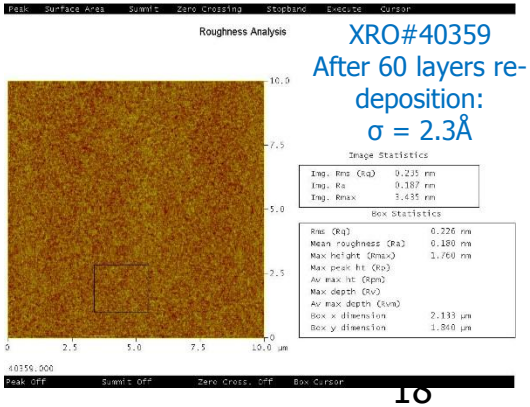




XRO#40341
After 12 period removal:
σ = 5.3Å



XRO#40354
After 18 period removal:
σ = 3Å



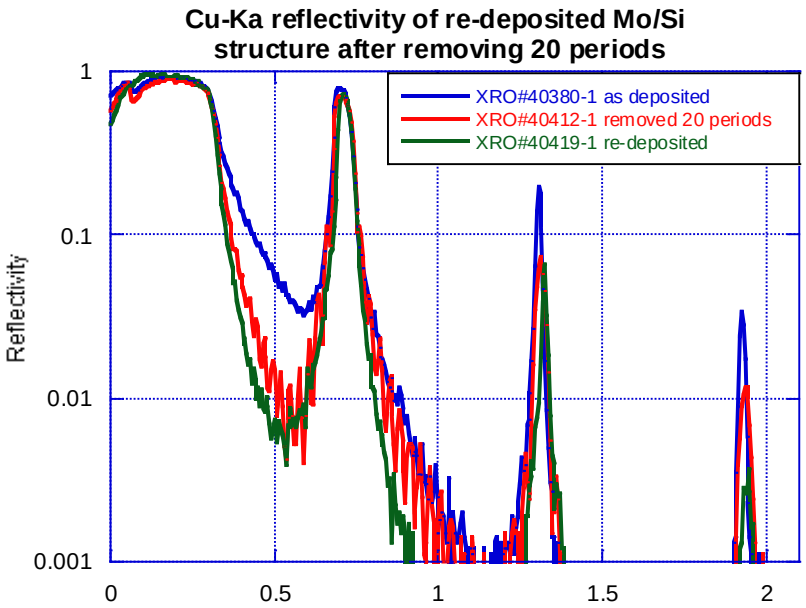
XRO#40359
After 60 layers re-deposition:
σ = 2.3Å

Full recovery – no smoothing needed

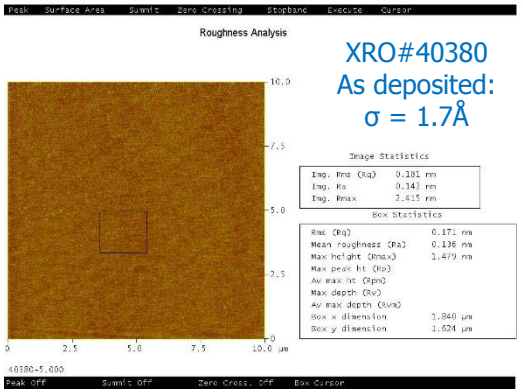
Ru capped Mo/Si ML – flats 1

Partial removal and re-deposition

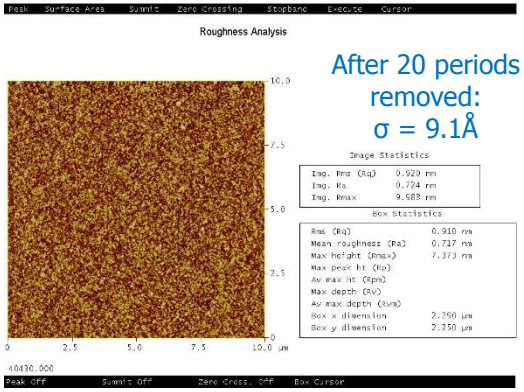
1. 40 periods of Ru-capped Mo/Si deposited
2. Ru layer and 20 top periods of Mo/Si ML removed by a plasma etching
3. 60 more periods deposited



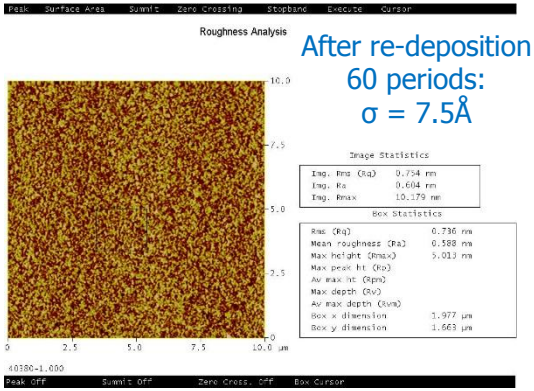
Very high roughness – smoothing required



XRO#40380
As deposited:
σ = 1.7Å



After 20 periods removed:
σ = 9.1Å

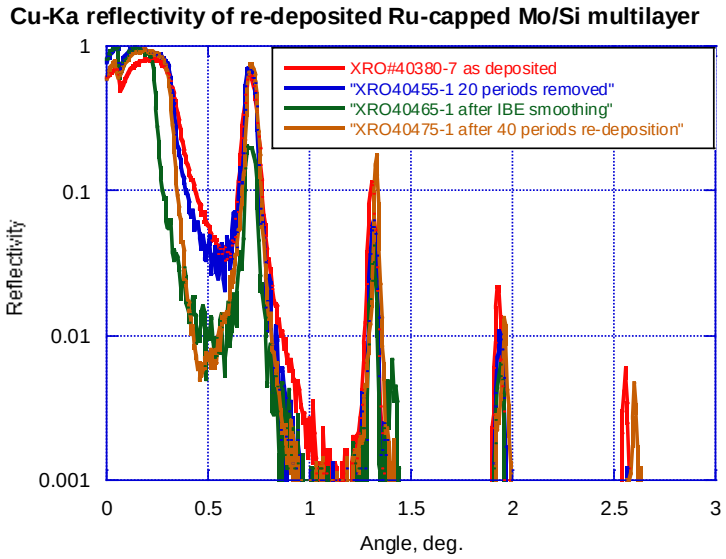


After re-deposition 60 periods:
σ = 7.5Å

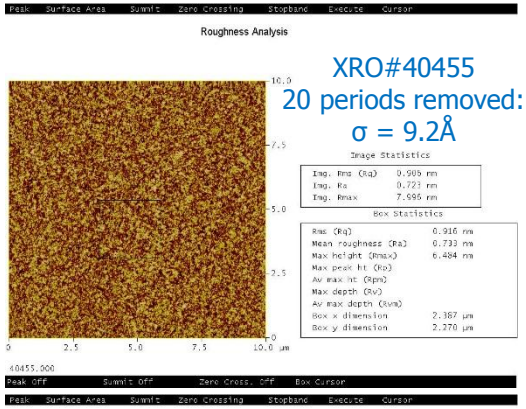
Ru capped Mo/Si ML – flats 2

Partial removal, smoothing by Ion Beam Smoothing process and re-deposition

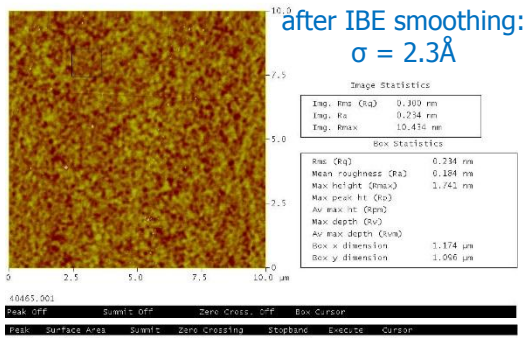
1. 40 periods of Ru capped Mo/Si deposited
2. Ru layer and 20 top periods of Mo/Si ML removed by a plasma etching
3. IB smoothing
4. 40 periods of Mo/Si re-deposited



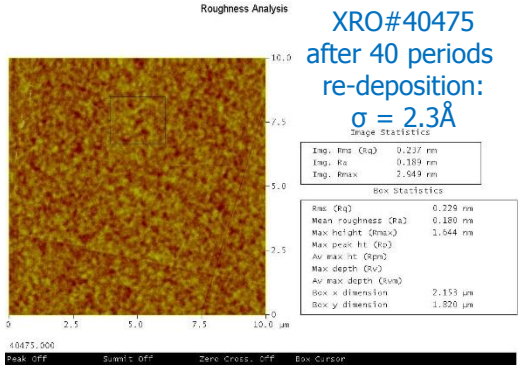
Fully recovered after IBS



XRO#40455
20 periods removed:
σ = 9.2Å



XRO#40465
after IBE smoothing:
σ = 2.3Å

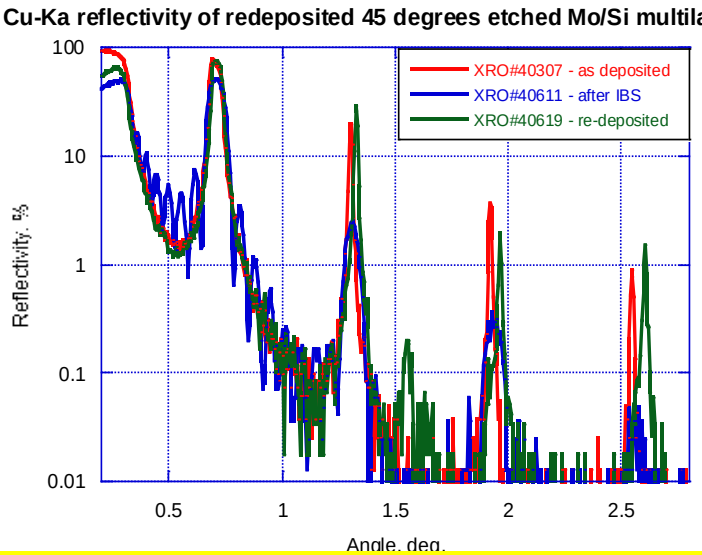


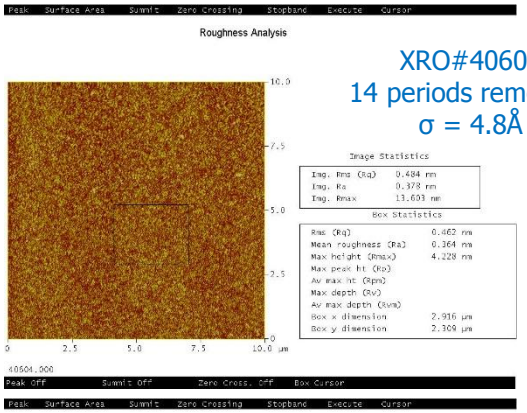
XRO#40475
after 40 periods re-deposition:
σ = 2.3Å

Plasma etching – curved sample

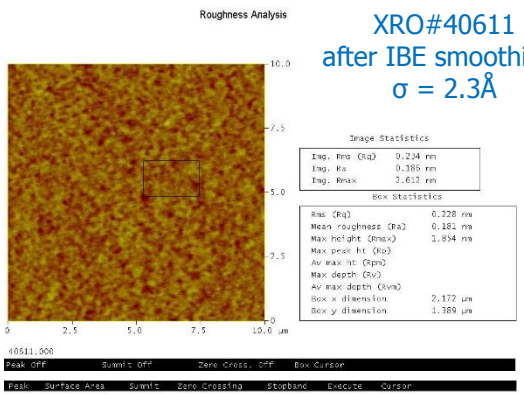
Plasma removal, smoothing by IBS and re-deposition

1. 40 periods of Mo/Si deposited
2. 14 top periods of Mo/Si ML removed by a plasma etching
3. IB smoothing
4. 40 periods of Mo/Si re-deposited

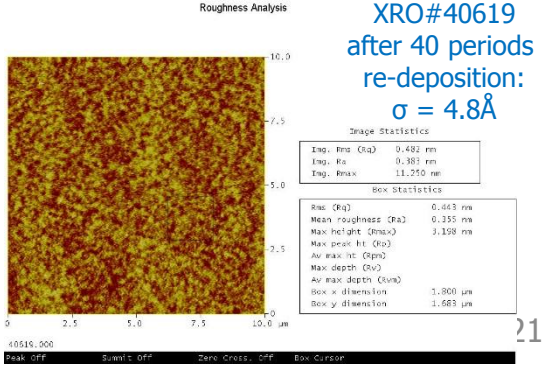




XRO#40604
14 periods removed:
σ = 4.8Å



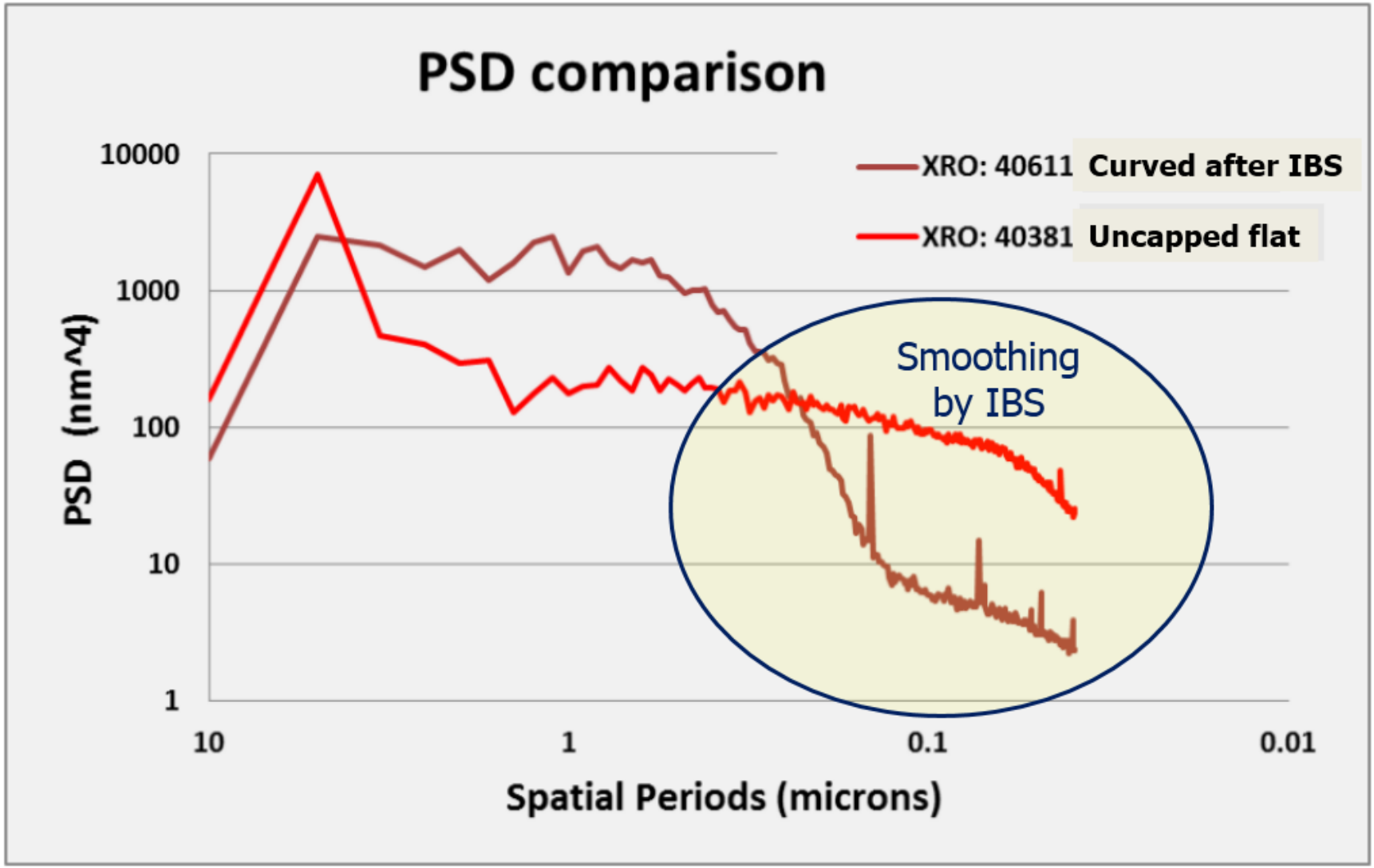
XRO#40611
after IBE smoothing:
σ = 2.3Å



XRO#40619
after 40 periods
re-deposition:
σ = 4.8Å

AFM roughness is high after re-deposition. Same conclusion is from Cu-K_α analysis. R(EUV)=?

Roughness: flat vs curved



Conclusions

- SiO_2 and TiO_2 capping layers: demonstrated practically a full oxidation.
- ZrO_2 capping layers: all Zr is bound to O with ~ 15% – 25 % in form of zirconium carbonate.
- ZrO_2 capping layer was improved since February'15 and now both TiO_2 and ZrO_2 show a similar reflectivity loss after EUV exposure
- Wet etching successfully removes tin without effecting performance of Mo/Si ML coating
- Wet etching approach should work to strip ML from a collector optics but the process is quite messy
- Plasma etching works well on flat optics but it results in surface roughness increase on curved optics
- Ion Beam Smoothing process reduces surface roughness after plasma etching but a further reduction is still needed

Thank you

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